MODIFIED PALM OIL FUEL ASH (POFA) AS QUARTZ REPLACEMENT FOR THE PRODUCTION OF PORCELAIN

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DEDICATION

This thesis is specially dedicated to my parents especially my beloved mother Amina Muhammad for her support, prayer, encouragement and unconditional love, may God almighty reward you.

To my beloved wife Sadiya Yusuf Haruna, my kids Abdallah Sani Durumin iya and AbdurRahman Sani Durumin iya and my brothers.

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ABSTRACT

Utilization of silica rich ash such as palm oil fuel ash (POFA) in the ceramic industry due to the increasing demand for porcelain with improved performance is inevitable. Wastes are produced from the industry in form of empty fruits bunches, kernel and fibers. Palm oil industries burned the waste to produce heat to the boiler and generate electricity, the ash produced is known as palm oil fuel ash. POFA has similar chemical property with quartz, thus can be used as quartz replacement. The objectives of this research are to modify POFA, determined its effect on physico-mechanical properties of porcelain and develop a formulation model. To remove the moisture of POFA, the powder was dried in an oven at 110 °C for 24 hours and ground to a sufficient fineness $\leq$ 50 μm for 12 hours at 250 rev/min. POFA powder underwent heat and HCL acid treatments. Standard porcelain of 50 % clay, 25 % feldspar and 25 % quartz was adopted. Quartz was substituted with POFA at 0, 15, 20 and 25 wt.% and mixed homogeneously with the composition of porcelain, dry pressed into pallets at 91 MPa and sintered at 1100 °C, 1150 °C, 1200 °C and 1250 °C for 2 hours soaking time. Modified POFA (SiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, and P_2O_5) were then added to the optimum composition (1150 °C, 15 wt.% of POFA and 2 molar HCl acid treatment) at 1, 2, 3, 4, 5, 10 and 15 wt. % mixed homogeneously, dry pressed and sintered at 1150 °C to identify their effect on physico-mechanical properties of porcelain. Densification was achieved at lower sintering temperature by addition of POFA. Due to formation of mullite and crystalline phase, the highest values of bulk density, compressive strength, and Vickers micro hardness were found to increase by addition of Fe_2O_3 at 5 wt. % as 2.515 g/cm^3, 177.08 MPa and 829 HV respectively. The results of developed polynomial regression models show a very good prediction similar to the experimental value. Hence, POFA has a greater future in ceramic industry due to its flexibility and chemical properties. It is therefore evidently concluded that, addition of Fe_2O_3 at 5 wt. % enhanced both physical and mechanical properties of porcelain.

ABSTRAK

Penggunaan abu yang kaya dengan silika seperti abu bahan api kelapa sawit (POFA) dalam industri seramik kerana peningkatan permintaan terhadap porselin dengan peningkatan prestasinya tidak dapat dielakkan. Sisa buangan dihasilkan dari industri ini dalam bentuk tandan buah kosong, kernel dan serat. Industri minyak sawit membakar sisa buangan itu untuk menghasilkan haba kepada dandang dan menjana elektrik, abu yang dihasilkan dikenali sebagai abu bahan api sawit. POFA mempunyai sifat kimia yang sama dengan kuarza, oleh itu ia boleh digunakan sebagai pengganti kuarza. Objektif penyelidikan ini adalah untuk memodifikasikan POFA, menentukan kesannya terhadap sifat-sifat fizikal-mekanik porselin dan membangunkan model perumusan. Untuk menghilangkan kelembapan POFA, serbuk itu dikeringkan di dalam ketuhar pada 110 °C selama 24 jam dan dikisar ke kehalusan yang mencukupi ≤ 50 μm selama 12 jam pada 250 rev/min. Serbuk POFA dikenakan rawatan haba dan asid HCL. Porselin piawai 50% tanah liat, 25% feldspar dan 25% kuarza telah diterima pakai. Kuarza digantikan dengan POFA pada 0, 15, 20 dan 25 % berat dan dicampurkan secara homogen dengan komposisi porselin, dipaletkan secara kering pada 91 MPa dan disinter pada 1100 °C, 1150 °C, 1200 °C dan 1250 °C untuk 2 jam masa rendaman. POFA yang dimodifikasi (SiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, dan P_2O_5) kemudian ditambahkan kepada komposisi optimum (1150 °C, 15 % berat POFA dan 2 molar rawatan asid HCl) pada 1, 2, 3, 4, 5, 10 dan 15 % berat campuran homogen, dipaletkan secara kering dan disinter pada 1150 °C untuk ditentukan kesannya terhadap sifat-sifat fizikal-mekanik porselin. Densifikasi dicapai pada suhu pensinteran yang lebih rendah dengan penambahan POFA. Oleh kerana pembentukan mullite dan fasa kristal, nilai tertinggi ketumpatan pukal, kekuatan mampatan, dan kekerasan mikro Vickers didapati meningkat dengan penambahan Fe_2O_3 pada 5 % berat masing-masing pada 2.515 g/cm^3, 177.08 MPa dan 829 HV. Hasil model regresi polinomial yang dibangunkan menunjukkan ramalan yang sangat baik seperti nilai eksperimen. Oleh itu, POFA mempunyai masa

depan yang lebih besar dalam industri seramik kerana fleksibiliti dan sifat kimianya. Dengan ini jelas terbukti bahawa penambahan Fe_2O_3 pada 5 % berat meningkatkan sifat fizikal dan mekanik porselin.

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LIST OF SYMBOLS AND ABBREVIATIONS

A	-	Area
d	-	Distance
F	-	Force
n	-	Integer
α	-	Alpha
β	-	Beta
θ	-	Angle
λ	-	Wavelength
π	-	Pi
$<$	-	Less than
W_1	-	Dry Weight
W_2	-	Suspended Weight
W_3	-	Soaked Weight
ρ_w	-	Density of Water (0.99777 g/cm^3)
ASTM	-	American Society for Testing and Materials
EDX	-	Energy Dispersive X-ray
FA	-	Fly Ash
HF	-	Hydrofluoric acid
ISO	-	International Standards Organization
ICDD	-	International Centre for Diffraction Data
OPC	-	Ordinary Portland cement
POFA	-	Palm Oil Fuel Ash
RHA	-	Rice Husk Ash
SEM	-	Scanning Electron Microscopy
XRF	-	X-ray Fluorescence
XRD	-	X-ray Diffraction

FFB	-	Fresh Fruit Branches
OER	-	Oil Extraction Ratio
POFA	-	Palm Oil Fuel Ash
EFB	-	Empty Fruit Bunches
CS	-	Ceramic Sludge
OPS	-	Oil Palm Shell
SCM	-	Supplementary Cementing Materials
SA	-	Sawdust Ash
SF	-	Silica Fume
SiC	-	Silicon Carbide
SCC	-	Self-Compacting Concrete
CPH	-	Close-packed Hexagonal arrangement
IUPAC		International Union of Pure and Applied Chemistry
HCl	-	Hydrochloric Acid
AM	-	Additive Manufacturing

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Malaysia is among the major crude palm oil supplier in the world. It is estimated that, in Malaysia there is 5.64 million hectares of palm oil stations, and states of Sabah and Sarawak alone has about 1.54 and 1.44 million hectares (which is 37 % and 26 % of the total planted area) that is, it has the largest planted area in whole Malaysia. Malaysia produced approximately 15 million tonnes of crude palm oil in 2006 alone, annually the estimated production of fresh fruit branches (FFB) is around 75 million tonnes with the average oil extraction ratio (OER) of about 20 % (Nambiappan et al., 2018)

In Malaysia, palm oil industries are the predominant agro-industries. Palm oil industries burn palm oil shells, empty fruit bunches and fibers as fuel to heat up boiler and generate electricity, the by-product disposed is known as palm oil fuel ash (POFA) (Bamaga et al., 2013).

Palm oil mills industry in 2006 revealed that during the crude palm oil production, a significant amount of waste materials are produced such as shells of palm oil, empty fruit bunches (EFB) and fibers (husk). It is also estimated that, for every 100 million tonnes of crude palm oil processed, million tonnes of wastes is disposed with no commercial value and environmental use, these wastes normally can be reutilized as potential cement replacement materials especially in the concrete and construction industries (POBM, 2006).

The recycling of these waste materials such as POFA, rice husk ash (RHA) and ceramic sludge (CS) is very useful, POFA as one of the pozzolanic materials (a siliceous and Aluminous material which in the presence of water react chemically with $Ca(OH)_2$ to form cementitious material) that have been used for many applications such as cement replacement. Pozzolanic materials are fine materials, which contain silica, alumina in higher amount and calcium oxide, iron oxide, magnesium oxide and alkalis in fewer amounts. Silicon oxide (SiO_2) is a major composition of POFA, when silica and alumina react in the presence of calcium oxide (CaO_2) or calcium hydroxide $Ca(OH)_2$ there is formation of cementations materials which can be used as a cement replacement and can exhibit high strength in concrete more than the normal cement, as the particle size of POFA reduced, so the strength is increased (Patel & Shah, 2015).

Based on this, effective use of POFA as a replacement in the cement production and in the ceramic companies will reduce the environmental problem and also reduce cost of production in both companies. According to Karim et al., (2011) the use of POFA in concrete as cement replacement is logical, worthy and good for the concrete industry especially in the present situation for the reduction of cost of construction material and minimization of waste disposal problem.

Porcelain are extensively used as traditional ceramics for applications such as scientific, household and engineering. Traditionally, porcelain consists of clay (for plasticity during formation), feldspar (as fluxing agent, it react with amorphous silica to influence glass formation) and quartz (filler). Porcelain products have many advantages such as low water absorption, durability, translucence and high mechanical strength (Lerdprom et al., 2017).

According to Prasad and o-authors properties of porcelain can be improved by adding the rice husk ash that has very small amount of carbon incorporated in the white-ware structure. Hence, the flabby structure of rice husk ash that unfavorably affect the final result of the porcelain body, and also lead to increase of the loss as a result of cracking and body deformation. By replacing quartz with palm oil fuel ash (POFA) or rice husk ash (both ashes have SiO_2 as major chemical compounds and are pozzolanic in nature), the mechanical strength of porcelain is expected to increase, other wastes that act as quartz replacement are; kyanite, bauxite, fly ash, sericitic pyrophyllite ($Al_2Si_4O_{10}(OH)_2$) and silliminite sand (Al_2SiO_5) (Prasad et al., 2003).

1.2 Problem statement

It is estimated that Malaysia supply 44 % of the total palm oil to the world (MPOB, 2017). Hence, Malaysia has become among the main producers of palm oil, and this figure is expected to increase annually (Chandara et al., (2010); Jamo et al., (2014); Prasad et al., (2003)). Therefore, due to the amount of POFA disposed frequently research should be intensified on how to properly manage this waste and appropriately used it (Chandara et al., 2010).

To mitigate the waste disposed from oil palm industries, some research have been conducted on how to use POFA in industries such as construction and ceramic industry (as quartz replacement). It is revealed that POFA is a good pozzolanic and therefore can be used as an additive in the production of cement for medium particle size and small particle size to a certain level up to 20 % and 30 % (Jamo et al., 2014).

It was reported by Momeen et al., (2016), 5 million hectares of land were used for oil palm plantation in Malaysia. Due to this vast area resulted in the annual production of about 61.1 million ton of solid waste, like empty fruit branches, fibers, and kernels in the country (Kang et al., 2013). These waste materials are dumped around the industries after the palm oil extraction and posed severe land pollution. As such, there is a great prospect to utilize waste materials from the palm oil industry such as palm oil shell (POS), palm oil fuel ash (POFA) as a replacement for conventional materials in the production of porcelain and concrete (Momeen et al., 2016). POFA is the by-product obtained from the burning of wastage from the palm oil industries such as empty fruit bunches, POS and fibers at temperatures between 800 °C to 1000 °C as fuel to generate electricity in palm oil mills (Megat et al., 2012). A Huge amount of POFA was disposed each year as waste with limited utilization, and this could cause a health hazard and environmental problems. Research on utilization of POFA as cement replacing materials dated back to 1990s. According to literature, original ungrounded POFA (POFA without grinding) is less effective as supplementary cementing materials (SCM) due to its larger size and being porous produces weak microstructure (Safiuddin et al., 2011).

Therefore, researchers began utilizing grinded POFA, which had a better microstructure and higher reactivity to act as a more effective pozzolanic material (Megat et al., 2012). Porcelain production have recently been the major breakthrough made by the ceramic industries, the production is quite similar to traditional ceramics, but porcelain is very dense materials that after sintering at a temperature between 1100 °C to 1215 °C, it is characterized with glassy phase of mullite and quartz crystals. Subsequently, research shows that the major chemical composition of POFA is quartz (Esposito et al., 2005).

Due to high demand of flexibility in porcelain production ranging from customizing, prototyping to several series of art, researchers reported a difficulty in producing an intricate porcelain shapes (Lima et al., 2018). Zocca et al., (2015) suggest the use of additive manufacturing (AM), such as palm oil fuel ash (POFA), fly ash (FA) and rice husk ash (RHA) to enable solve the pressing challenge of shape modification. The researchers further revealed that, incorporation of AM enable innovative shaping with the possibility of modifying the traditional porcelain production.

Production of porcelain is faced with economic and technical difficulties as result of high demand for high purity in the raw materials and need for enriched glassy phase required to produce less porous and low water absorbing porcelain, this forced researchers and industries to investigate alternative raw materials such as palm oil fuel ash (POFA) to be used in the production of porcelain (Ediz & Yurdakul, 2009). Several research have reported the use of wastes and ashes such as fly ash, rice husk ash and palm oil fuel ash (POFA) to enhance mechanical and physical properties of porcelain (Dana et al., (2005); Esposito et al., (2005); Fernandes & Ferreira, (2007); Ferrari & Gualtieri, (2006); Gennaro et al., (2003 & 2007); Luz & Ribeiro, (2007); Raimondo et al., (2007); Rambaldi et al., (2007); Tucci et al., (2004)).

Hence, most of the research available on utilization of POFA so far emanate from civil engineering or construction industries, POFA is either used as partial replacement of cement in concrete or it is used as additives in Asphalt, only few were from ceramic industries where POFA is used as quartz replacement, this indicate the use of POFA in porcelain is a new development in the ceramic industry.

A research conducted by Jamo et al., (2014), on mechanical properties of ceramic tiles by replacement of quartz with rice husk ash and POFA is among the few research available that focused purely on ceramic. The researchers were able to show that, the compressive strength increase was due to substitution of quartz with rice husk ash and POFA. Therefore, this research focus on the modification of

POFA, its effect on physico-mechanical properties of porcelain and also formulate equation for optimum physico-mechanical properties of porcelain.

1.3 Objectives of study

The objectives of this research are;

i. To optimize the method of POFA preparation/modification to be used as quartz replacement.
ii. To investigate the effect of substitution of quartz with POFA on porcelain production and determine role of modified POFA on physico-mechanical properties of porcelain.
iii. To develop a model that optimize physical and mechanical properties of porcelain.

1.4 Significance of study

The research aimed at identifying the suitable method for the preparation/modification of POFA, the research will also determine the effect of substitution of modified POFA on physico-mechanical properties of porcelain. Thus, this research is very important as it will a model in achieving optimum physical or mechanical properties of porcelain. Consequently, result of this research will help the industries cut the cost of producing porcelain by suggesting a replacement of quartz with the more abundant element which is extracted from waste product POFA. The research is also aimed at being a tool to make the environment friendly by suggesting ways of using this organic waste POFA that previously ended up in a landfill and was disposed of no industrial use. Lastly, the research is significant as it serves as media whereby Malaysian government will use the outcome for revenue generation by modifying this waste POFA and sell to the ceramics industries locally and internationally in this century of global financial crisis.

1.5 Scope and limitations of study

To achieve the objectives of this research the following scopes were adopted;

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